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QUALITATIVE DETERMINATION FOR DEGREE OF DECOMPOSITION OF COMPOST BY THE STARCH-IODINE METHOD

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This is a preliminary report on the starch iodine method for determining the degree of decomposition in municipal wastes, or compost. All refuse contains starch which must be degraded before refuse becomes a microbiologically stable product. Testing for its presence will indicate whether or not the compost has stabilized. The test is based on the formation of the iodine-starch complex in an acidic extract of compost and has the following advantages: It is rapid and easily performed, it is specific for starch and the equipment is very simple. This is only a qualitative spot test designed to show relative change. After checking satisfactory and unsatisfactory compost with this test, finished compost always gives a characteristic color reaction (yellow).

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A Preliminary Report

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Joint U.S. Public Health Service--Tennessee Valley Authority Composting Project
Johnson City, Tennessee

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Richard D. Lossin*

A simple and rapid test has long been needed to determine the extent of decomposition of compost. Many tests have been proposed and used, including the measurement of the reduction in total carbon or the carbon-nitrogen ratio, increase in percent ash, decrease in cellulose, and decrease in lipids. These methods are quite adequate to determine the completeness of composting, assuming a representative sample has been taken. All of them, however, have obvious disadvantages; each requires complicated sample preparation, considerable time, and, in most cases, expensive equipment.

Microorganisms degrade the most readily available substrates in municipal refuse to yield a stable, humus-like product, that gardeners call compost. Starch has always been found in measurable quantities (2 to 6 percent) in municipal refuse by this investigator, and, since starch is in the class of easily degraded substrates that must be broken down and metabolized before the refuse becomes a microbiologically stable product, testing for its presence will indicate whether or not the compost is stabilized. The rationale of the test rests on the hypothesis that all refuse contains a measurable quantity of starch and that the starch must

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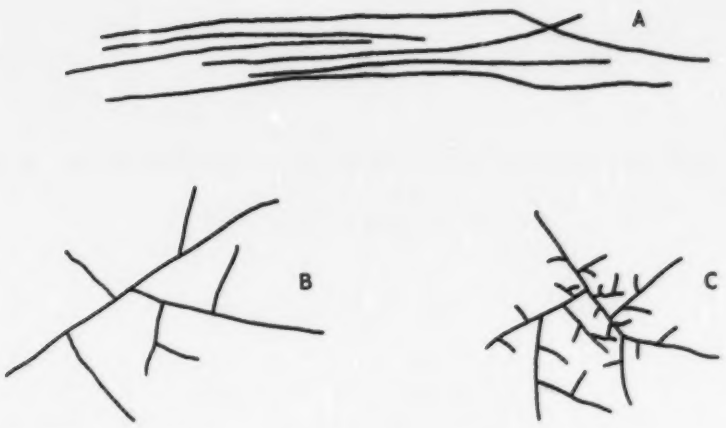

FIGURE 1. Amylose (A), amylopectin (B), and glycogen (C)

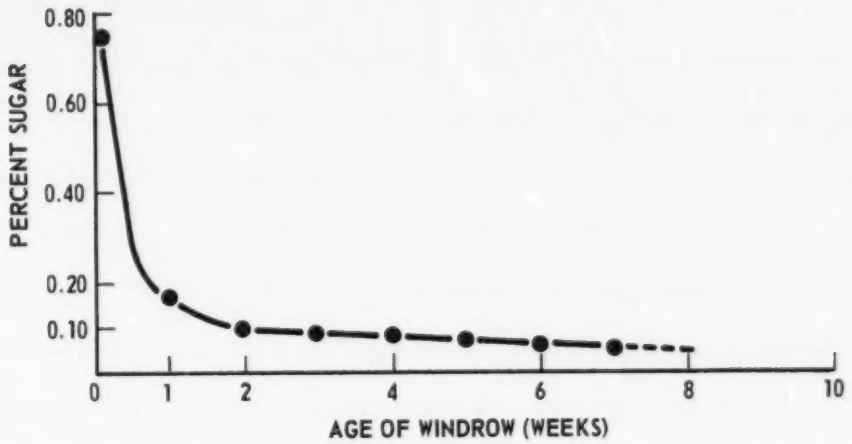

FIGURE 2. Decrease of sugar versus age of compost.

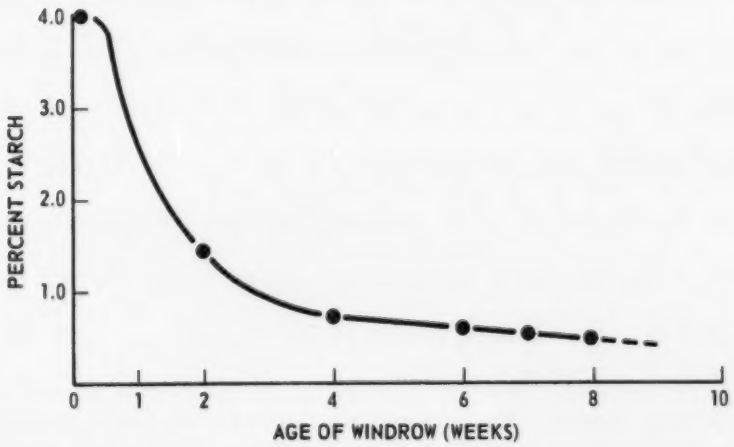

FIGURE 3. Decrease of starch versus age of compost.

be degraded before the compost can be considered acceptable, no matter how much was initially present. The test is based on the formation of the iodine-starch complex in an acidic extract of compost and has the following advantages: it is rapid and easily performed, it is specific for starch, and the equipment necessary is very simple.

Although the method has not been tested on different composts, it was valid for compost from the Joint Public Health Service--Tennessee Valley Authority Composting Project in Johnson City, Tennessee. The method is presented here to aid other workers in the field and to further test the method.

Theory

Three types of carbohydrates are found in compost: sugars, starch, and cellulose (Figure 1). These constituents decrease with age of the compost (Figures 2 to 4). Sugars are metabolized first, being almost consumed within a week; starches are next, followed by cellulose. After about 4 to 5 weeks of windrow composting, the starch has passed through its maximum rate of degradation. This is the point where the compost first has an acceptable appearance and odor, has gone through its maximum temperature phase, and has reached its maximum pH (Figures 5 and 6). In other composting processes with different types of refuse, these events may occur at different time intervals, but the relationships can be expected to be approximately the same.

FIGURE 4. Decrease of cellulose versus age of compost.

FIGURE 5. Temperature versus age of compost.

Certain polysaccharides (starches: amylose, amylopectin, glycogen, and dextrans) form characteristic color complexes when combined with molecular iodine.[1-3] Amylose (straight-chain starch) gives an intense blue-black color; amylopectin (moderately branched) gives from light blue to purple to red colors, depending on the degree of branching; and glycogen (highly branched) gives reddish-brown colors. Dextrans, which are partial degradation products of starch, give reddish-brown to yellow colors. The hydrolysis of starch may be followed by the gradual change in the iodine complex color (blue-black to light blue to purple to red to yellow to none).

The branching varies with different starches. The most highly branched starches have the least affinity for iodine, as do the shortest chains of starch (Figure 7). Evidence to date indicates that an adsorptive complex is formed between iodine and the helically coiled polysaccharide chains. Due to the α, 1-4 glycosidic linkages in the molecule, amylose is not linear. The conformational strain imposed by the alpha-linkages coils the chain into a helix.[4] Comparison to benzamide complexes of iodine ($HI_3 \cdot BzNH_2$) indicates that the starch complex contains triiodide ion (I_3^-) chains linked end to end.[1] In 1957, Hollo concluded from the heats of formation that the iodine is hydrogen-bonded to the amylose chain: the glucopyranoside rings apparently turn with their edges carrying trans-hydroxyl groups toward the iodine molecules.[5] Circular dichroism studies of the iodine-starch complex

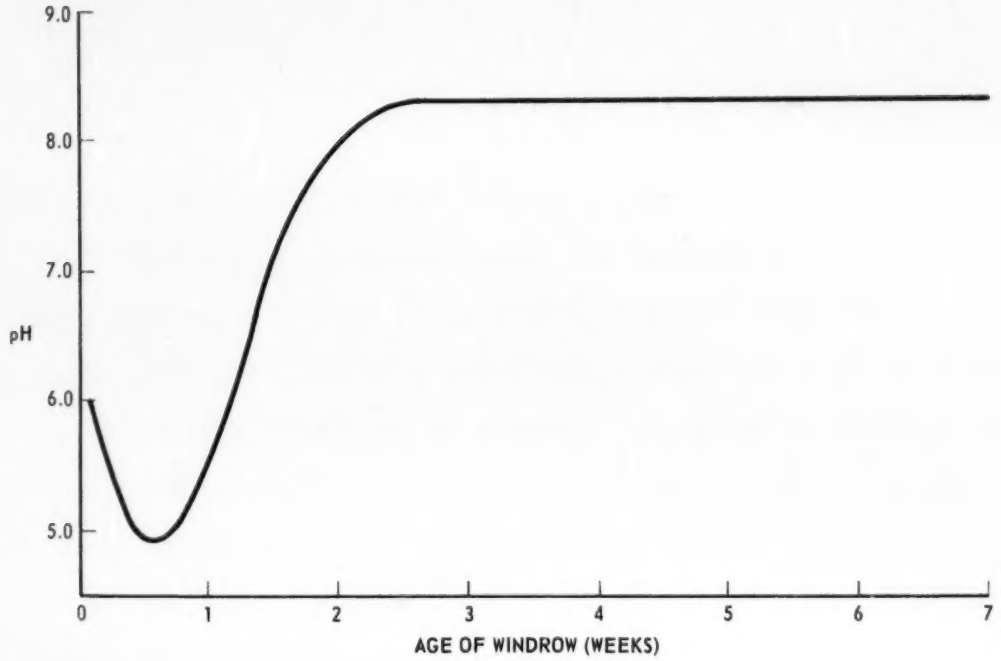

FIGURE 6. pH versus age of compost.

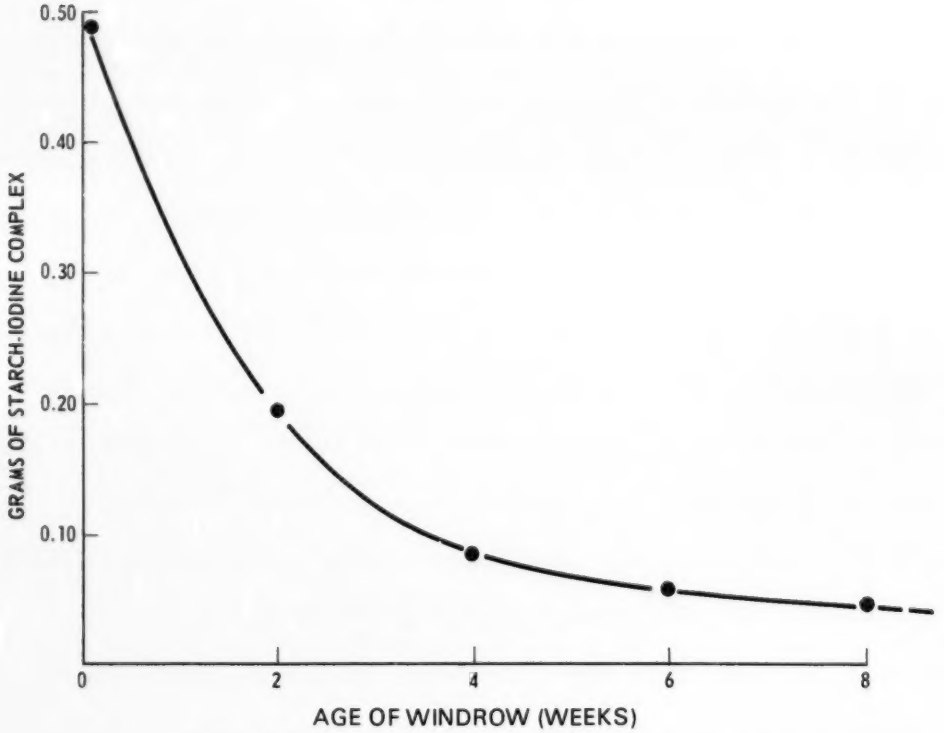

FIGURE 7. Complex recovery with age of compost.

suggested that a six-unit repeating helix is present with one iodine molecule in the middle.[6] By means of potentiometric titrations of the iodine-amylose complex, Eliassaf and Lewin concluded that one molecule of iodine was bonded to 6.8 ± 0.2 aldoglucoside units.[7] They also found the equilibrium constant of the complex to be 2.4×10^4 at 25°C. Miyazaki found the color of the complex to be unaffected by strong acid, but that raising the pH above 7.0 decreased the color.[8] He also found the color to be intensified by the presence of KCl.

In the present study a test was run to determine whether all the starch found in the compost was complexing with iodine. The weight of starch-iodine complex recovered from 10-g compost samples varied with different compost ages (Figure 7). This corresponded very well with determinations of the amount of starch in the compost (Figure 3).

Procedure

The iodine reagent is prepared by dissolving 2.00 g KI in 500 ml H_2O, then dissolving 0.80 g I_2. The other reagent is perchloric acid (36%).

Place about one gram compost (*see* Discussion) in a 100-ml beaker, wet with a few drops of ethanol if dry, add 20 ml perchloric acid (36%) and stir. Filter through open-texture filter paper (e.g., Whatman No. 90). Add 2 ml iodine reagent to the filtrate and stir. Place a few drops on a spot plate or other white background, then note the

color and precipitate. Finished compost will give yellow color and very little precipitate. Poor or unfinished compost will give dark blue color and heavy blue precipitate. The color sequence normally found as the age of compost increases is blue-black → light blue → grey → green → yellow. A reddish color may be found with partially degraded compost.

It is possible that either little or no color will be produced by the test described or, more likely, tests on all samples for the entire compost cycle will be too dark. There are five variables present in this test, one or more of which are responsible for these deviations: (1) starch concentration and type, (2) particle size of compost, (3) sample size, (4) iodine concentration, (5) perchloric acid concentration.

The method has been checked on a variety of samples by altering all of these variables and the following comments reflect the results.

The iodine concentration must be in excess of that needed so as not to become a limiting factor. If all the ages of compost give a color reaction so dark that it is difficult to differentiate between them, one or more of the following steps may be taken. (1) Reduce the perchloric acid concentration. Concentrations down to 5 percent were found to give positive tests with unfinished compost. (2) Dilute the filtrate before adding the iodine reagent. (3) Reduce the sample size.

Step 3 is not advisable, as a reduction in sample size will increase the chance of a nonrepresentative sample. It is best to have

the sample as large and as finely ground as possible. Step 1 is preferable, with Step 2 being next. If the color is too light, then the best policy is to increase the sample size or grind the sample more finely. Do not increase the perchloric acid concentration. When standardizing the test, be sure to use finished and unfinished compost as standards, making certain that the variables are adjusted to give a blue color with unfinished compost and a yellow color with finished compost. Once these variables are established, they must not be altered. Do every test in exactly the same fashion.

Discussion

One must remember that this is only a qualitative spot test, designed only to show a relative change; nothing is being quantitatively determined. The test is to be used only as a control measure to check the completeness of composting in one particular process. After checking satisfactory and unsatisfactory compost with this test, it will become apparent that finished compost always gives a characteristic color reaction (yellow), whereas unfinished compost will not. Finished compost has never given a positive starch reaction with this test.

It is difficult to obtain a representative sample from compost because of its nonhomogeneity. In order for the test to be valid, it is necessary to work with a representative sample; that is, the overall characteristics of the sample must be similar to the part being sampled. To this end, the following sampling procedure is outlined:

From the digestor or windrow select small grab samples (50-100 g) randomly to make a total composite sample of at least 1 kg, the larger the sample the better. This sample should be rough ground, (by W-W grinder,* hammermill, etc.) and thoroughly mixed; duplicate samples are taken from this for testing. In general, the representative character and homogeneity of the sample are improved by the following: (1) increasing the sample size, (2) taking a larger number of composites; (3) finer grinding; (4) better mixing.

There is always a finite probability, however small, of selecting a sample at any stage of the composting cycle that will contain excessive starch or no starch at all. The reliability of the testing procedure can be checked by taking several different samples from the same sample population (compost pile) and performing the same test on all of them. If they are not all nearly the same, then the sampling technique should be modified to correct any gross fluctuations.

*Mention of a commercial product does not imply endorsement by the U.S. Public Health Service.

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